Age of the oldest Homo sapiens from eastern Africa

Céline M. Vidal, Christine Lane, Asfawossen Asrat, Dan N. Barfod, Emma L. Tomlinson, Amdemichael Zafu Tadesse, Gezahegn Yirgu, Alan Deino, William Hutchison, Aurélien Mounier, Clive Oppenheimer

Methods

Stratigraphic descriptions and sampling were carried out during two field seasons in 2017 and 2018. We sampled the Qi2 eruption of Shala volcano previously described[1], and we revisited Konso [2,3], Omo-Kibish [4–6], and Gademotta [7,8] formations (Figure 1). At each site we described extensively the stratigraphy of the outcrops, measured the thickness of units and sampled deposits where best exposed and least altered.

$^{40}Ar/^{39}Ar$ dating

Feldspars were extracted from pumice samples at the Departments of Geography and Earth Sciences, University of Cambridge. Rocks were crushed in a jaw crusher and sieved to obtain a 250–500 μm size fraction, cleaned under water, and passed through a Frantz magnetic barrier laboratory separator to isolate sanidine phenocrysts from the groundmass. Because separates would still contain other phases (primarily glass and quartz), 100-200 sanidine grains were further handpicked and then leached in 5% HF to remove any glass attached to the crystals.

Samples and neutron flux monitors were packaged in copper foil and stacked in quartz tubes with the relative positions of packets precisely measured for later reconstruction of neutron flux gradients. The sample package was irradiated in the Oregon State University reactor, Cd-shielded facility. Alder Creek sanidine (1.1891±0.0008 (1σ) Ma (ref. [9]) was used to monitor ^{39}Ar production and establish neutron flux values (J) for the samples. Gas was extracted from samples via step-heating using a mid-infrared (10.6 μm) CO_2 laser with a non-gaussian, uniform energy profile and a 1.5 mm beam diameter. The samples were housed in a doubly-pumped ZnS-window laser cell and loaded into a stainless steel planchette containing 208 2.0mm diameter round wells. Liberated argon was purified of active gases, e.g., CO_2, H_2O, H_2, N_2, CH_4, using three Zr-Al getters; one at 16°C and two at 400°C. Data were collected on a Mass Analyser Products MAP-215-50 single-collector mass spectrometer using an electron multiplier collector in dynamic collection (peak hopping) mode. Time-intensity data are regressed to inlet time with second-order polynomial fits to the data. The average total system blank for laser extractions, measured between each sample run, were 17.0±0.7×10-16 mol ^{40}Ar, 19.6±0.4×10-18 mol ^{39}Ar, and 7.8±1.6×10-18 mol ^{36}Ar (Table S5). Mass discrimination was monitored on a daily basis, between and within sample runs by analysis of an air standard aliquot delivered by an automated pipette system (see raw data for D values applied to individual steps in Table S4). All blank, interference and mass discrimination calculations were performed with the MassSpec software package

(MassSpec, version 8.058, authored by Al Deino, Berkeley Geochronology Center). Decay constants and corrections are after Renne et al. 2011[10].

Peak age distributions were defined by determining the youngest population of individual grain analyses ($n \geq 10$) that conforms to a gaussian distribution with the expected scatter as indicated by the value of MSWD (Mean Square Weighted Deviation).

Ages for unit samples ETH17-14A1 and ETH17-14C are reported with two sigma errors in Table S2 with the raw data in Table S3.

Sample preparation for geochemical analyses

Sample preparation was carried out in the Cambridge Tephra Laboratory in-line with the protocols of the International Focus Group on Tephrochronology (INTAV)[11,12] for geochemical characterisation of volcanic glass. Pumice samples of the Qi2 Shala eruption were crushed, sieved at 500, 250, and 125 μm, and washed in purified water and hydrochloric acid (1%) in an ultrasonic bath. Glass grains from the 125-250 μm fraction were handpicked under microscope, mounted in epoxy resin stubs, then sectioned and polished. Distal tephra samples from Gademotta (Unit 10), Konso (TA-55/ETH18-14B and TA-56/ETH18-14A) and Kibish formations (KHS, ETH18-08) were washed through a sieve in purified water at 80 or 25 μm, then dried, described under microscope and mounted in epoxy resin stubs, then sectioned and polished. Strongly altered samples of TA-56 (ETH18-14A) and TA-55 (ETH18-14B) units from the Konso formation were density extracted to facilitate the search for volcanic glass[13,14]. Sample ETH18-14B from TA-55 was sieved at 125, 80 and 25 μm and residues inspected under the microscope, yet no glass was found.

Major element analysis

Mounted samples were analysed for major element compositions with a SX100 CAMECA electron microprobe at the Department of Earth Sciences, University of Cambridge, UK. Major elements were measured with an accelerating voltage of 10 kV, a 10 nA defocused beam. Elements were counted on-peak for 10 s (Na, Si), 20 s (Al, Fe and K), 60 s (Ti, Mg, Ca, and Cl), 90 s (P) and 120 s (Mn). Sodium was measured first in order to minimise alkali loss. The analytical accuracy was checked against international standards ATHO-G, STH-S6, VG-568 and internal peralkaline obsidian from Lipari, (74 wt% SiO_2, 3.8 wt% Na_2O, 5.3 wt% K_2O). Standards compositions are reported in Table S6. Where possible, we analysed at least 30 points per sample. All compositions are reported in Table S7.

Trace element analysis

Trace element compositions of individual tephra shards were analysed by LA-ICP-MS at the iCRAG laboratory at Trinity College Dublin. The instrument used was a Thermo iCAPQ coupled to a Photon Machines 193 nm G2 laser and a Helex two volume cell. We used a spot size of 40 μm, depending on

the area available for analysis, a repetition rate of 6Hz and a count time of 33 s (200 pulses) on the sample and 30 s on the gas blank (background). Concentrations were calibrated using NIST612 with ^{29}Si as the internal standard. Data reduction was undertaken in Iolite v3.4 and a secondary Ca correction factor was applied[15]. Accuracies of ATHO-G and StHs6/80-G MPI-DING glass analyses are typically better than 6% for most elements. Standards compositions are reported in Table S3 and detailed compositions are reported in Table S9.

References

1. Mohr, P., Mitchell, J. G. & Raynolds, R. G. H. Quaternary volcanism and faulting at O'A caldera, central ethiopian rift. *Bull. Volcanol.* **43**, 173–189 (1980).

2. Katoh, S. *et al.* Chronostratigraphy and correlation of the Plio-Pleistocene tephra layers of the Konso Formation, southern Main Ethiopian Rift, Ethiopia. *Quat. Sci. Rev.* **19**, 1305–1317 (2000).

3. Nagaoka, S. *et al.* Lithostratigraphy and sedimentary environments of the hominid-bearing Pliocene–Pleistocene Konso Formation in the southern Main Ethiopian Rift, Ethiopia. *Palaeogeogr. Palaeoclimatol. Palaeoecol.* **216**, 333–357 (2005).

4. Brown, F. H. & Fuller, C. R. Stratigraphy and tephra of the Kibish Formation, southwestern Ethiopia. *J. Hum. Evol.* **55**, 366–403 (2008).

5. McDougall, I., Brown, F. H. & Fleagle, J. G. Stratigraphic placement and age of modern humans from Kibish, Ethiopia. *Nature* **433**, 733–736 (2005).

6. Brown, F. H., McDougall, I. & Fleagle, J. G. Correlation of the KHS Tuff of the Kibish Formation to volcanic ash layers at other sites, and the age of early Homo sapiens (Omo I and Omo II). *J. Hum. Evol.* **63**, 577–585 (2012).

7. Morgan, L. & Renne, P. Diachronous dawn of Africa's Middle Stone Age: new 40Ar/39Ar ages from the Ethiopian Rift. *Geology* **36**, 967–970 (2008).

8. Laury, R. L. & Albritton, C. C. Geology of Middle Stone Age archaeological sites in the main Ethiopian Rift Valley. *Bull. Geol. Soc. Am.* **86**, 999–1011 (1975).

9. Niespolo, E. M., Rutte, D., Deino, A. L. & Renne, P. R. Intercalibration and age of the Alder Creek sanidine 40Ar/39Ar standard. *Quat. Geochronol.* **39**, 205–213 (2017).

10. Renne, P. R., Balco, G., Ludwig, K. R., Mundil, R. & Min, K. Response to the comment by W.H. Schwarz et al. on â€œJoint determination of 40K decay constants and 40Arâˆ—/40K for the Fish Canyon sanidine standard, and improved accuracy for 40Ar/39Ar geochronologyâ€• by P.R. Renne et al. (2010). *Geochim. Cosmochim. Acta* **75**, 5097–5100 (2011).

11. Lowe, D. J. *et al.* Correlating tephras and cryptotephras using glass compositional analyses and numerical and statistical methods: Review and evaluation. **175**, 1–44 (2017).

12. Lane, C. S., Lowe, D. J., Blockley, S. P. E., Suzuki, T. & Smith, V. C. Advancing tephrochronology as a global dating tool: Applications in volcanology, archaeology, and palaeoclimatic research. *Quaternary Geochronology* **40**, 1–7 (2017).

13. Blockley, S., Pyne-O'Donnell, S., … J. L.-Q. S. & 2005, U. A new and less destructive laboratory procedure for the physical separation of distal glass tephra shards from sediments. *Quat. Sci. Rev.* **24**, 1952–1960 (2005).

14. Lane, C. S., Cullen, V. L., White, D., Bramham-Law, C. W. F. & Smith, V. C. Cryptotephra as a dating and correlation tool in archaeology. *Journal of Archaeological Science* **42**, 42–50 (2014).

15. Tomlinson, E. L., Thordarson, T., Müller, W., Thirlwall, M. & Menzies, M. a. Microanalysis of tephra by LA-ICP-MS — Strategies, advantages and limitations assessed using the Thorsmörk ignimbrite (Southern Iceland). *Chem. Geol.* **279**, 73–89 (2010).